TECHNICAL MEMO 4

SUMMARY AND EVALUATION OF INTERIM QUARTZ TEST MATERIAL SAMPLE ANALYSIS BY SEM AND IR

1.0 INTRODUCTION

USEPA Region 8 is currently engaged in a program to test and evaluate a variety of analytical methods for quantification of asbestos in site soils, vermiculite insulation, and other related site samples. As part of this program, an initial pilot study was performed using a set of "interim quartz test materials" (IQTMs) with the aim of allowing a rapid initial assessment of the relative performance of infrared spectrometry (IR) and scanning electron microscopy (SEM) to quantify asbestos concentrations in the range of 0.01% to 1%. Quartz matrix was selected because it is judged to have negligible interference, maximizing the ability of each method to detect and quantify the added asbestos.

2.0 IQTM PREPARATION

Amphibole material used to spike the quartz matrix was obtained from a composite of ore samples collected from six locations at or near the Libby, Montana vermiculite mine site. The six samples were selected for this work because USGS analysis of their mineralogical composition found that they were highly enriched in asbestos (about 80% pure), contained the full range of amphibole types found at the Libby site, and were relatively free of interfering contaminants.

Samples were initially air dried in their original plastic bags and, when necessary, disaggregated to reduce the particle size to less than 3 cm in diameter. The six samples were then ground in a Hardinger horizontal grinder equipped with 3-inch diameter steel plates, producing material with a particle size less than 2 mm in diameter. The ground material was transferred to a 10 gallon drum fitted with a customized expanded steel mixing baffle. The drum was sealed and transferred to a horizontal roller apparatus where the sample was allowed to mix for a total of 24 hours. After mixing, the drum was relocated into a HEPA hood, and samples were removed for the final grinding step. In this grinding step, 200 g of amphibole material, 1 L of deionized water, and 200 ceramic grinding cylinders (2 cm x 1 cm) were transferred to a 5 L ceramic ball mill and the mill was sealed. The ball mill was transferred to the horizontal roller apparatus and allowed to grind the sample for a total of four hours. After grinding, the ball mill was relocated to the HEPA hood and the ceramic cylinders were removed and rinsed with deionized water. The ground amphibole was transferred from the ball mill into a plastic drying tray and the aqueous suspension was allowed to evaporate over several days. Once dry, the amphibole material was disaggregated using a metal spatula and transferred to a series of wide mouth one-liter glass jars and then sealed.

In the final preparation step, the ground amphibole material was wet mixed using an overhead mixing apparatus with 500 g of preground (<200 mesh) Brazilian quartz and then dried on a hot plate overnight in a HEPA hood. The dried material was transferred to a one-liter wide mouth glass jar fitted with a customized plastic mixing card. The container was sealed and transferred to a horizontal roller apparatus where the sample was allowed to mix for a total of 4 hours. Samples of the mixed material were then removed from the jar using a sample thief and transferred to a series (40) of one-ounce glass jars. A total of four IQTM samples were prepared in this manner representing a blank, 0.01, 0.1, and 1.0 weight percent amphibole spiking material (approximately 0, 0.008, 0.08, and 0.8% asbestos).

3.0 ANALYSIS

Eight IQTM samples were provided blind to each of two laboratories: EMSL Analytical, Inc. (EMSL), and Reservoirs Environmental Analytical Services (RESI). These samples are summarized in Table 1. EMSL analyzed samples by infrared spectroscopy (IR) and by scanning electron microscopy (SEM), while RESI analyzed the samples by SEM. The SOP for SEM is attached as Attachment 1. The SOP for IR is currently proprietary, and will be made available at a later date. A brief description of each method is provided below.

SEM

The SEM method involves examination of multiple fields of view using SEM at a series of different magnifications. At each magnification, the analyst records the area fraction of the field that is occupied by asbestos structures within a specified size range. Following completion of the analysis, the mass fraction is estimated using an equation that combines the results across each of the magnifications, assuming that area fraction and mass fraction are equivalent.

IR

In the IR method, the sample is spread out in a petri dish, and the IR spectrum is recorded at multiple locations across the surface of the sample. The concentration (mass percent) is estimated from the average of the multiple readings, using an empiric calibration curve. Because the IR method does not distinguish between massive and fibrous amphibole, all samples that are positive by IR are examined by PLM to determine if the response is due to massive rather than fibrous amphibole. In addition, all samples that are negative by IR are also screened by PLM as a double check that no visible asbestos is present.

It should be noted that the results of these pilot studies are expected to lead to improvements in the SOPs for one or both methods, and that the SOPs for these methods will be revised in the future, as appropriate.

4.0 RESULTS AND DISCUSSION

Table 1 lists the raw results for each sample for each laboratory. Results are discussed below, by method.

SEM Analysis

Figure 1 summarizes the SEM results from EMSL (upper panel) and RESI (lower panel) for the IQTM samples spiked with known amounts of asbestos by USGS. Inspection of Table 1 and Figure 1 reveals that both EMSL and RESI tended to overestimate the concentration of the sample containing 0.008% and 0.08% asbestos. EMSL tended to slightly underestimate the concentration for samples with 0.8% asbestos, while the results from RESI were slightly higher than the expected value.

Figure 2 provides an inter-lab comparison of SEM results from EMSL and RESI. The correlation coefficient (R) and the coefficient of determination (R^2) are 0.971 and 0.943, respectively. As noted in above, the agreement is good for all samples except the two with the highest concentration level.

IR Analysis

Figure 3 presents the results of IR analysis by EMSL. Note that the IR results are bounded between a lower reporting limit of 0.1% and an upper reporting limit of 1.0%. As seen, IR did not detect the 0.008% samples but did detect the 0.08% and 0.8% samples. Concentration values for the 0.08% and 0.8% samples estimated by IR were slightly high.

5.0 CONCLUSION

The data from this initial pilot study suggest that IR and SEM may be useful for quantification of asbestos levels in solid media. However, method performance in samples utilizing a quartz medium may not be representative of results that would be expected for asbestos in soil samples, so follow-up studies are needed using soil-based samples.

TABLE 1. SAMPLES AND RESULTS

Laboratory	Index ID	Nominal Mass % Total	Nominal Mass % Asbestos	Results (%) IR	Results (%) SEM
EMSL	GSC4000-9	0	0	< 0.1	< 0.01
	GSPP000-18	0	0	< 0.1	< 0.01
	GS4500K-20	0.01	0.008	< 0.1	0.08
	GSZ400G-14	0.01	0.008	< 0.1	0.02
	GSG0H0-16	0.1	0.08	0.1	0.22
	GSG2010-17	0.1	0.08	0.1	0.16
	GS02400-8	1	0.8	1	0.50
	GSO2100-12	1	0.8	1	0.61
RESI	GSD2000-14	0	0		ND
	GSH-9000-5	0	0		ND
	GSAB00C-6	0.01	0.008		0.03
	GS4S00N-23	0.01	0.008		0.03
	GSG2050-14	0.1	0.08		0.15
	GSG20C0-8	0.1	0.08		0.15
	GSO2100-11	1	0.8		0.96
	GSO-2100-5	1	0.8		0.96

FIGURE 1: SEM RESULTS

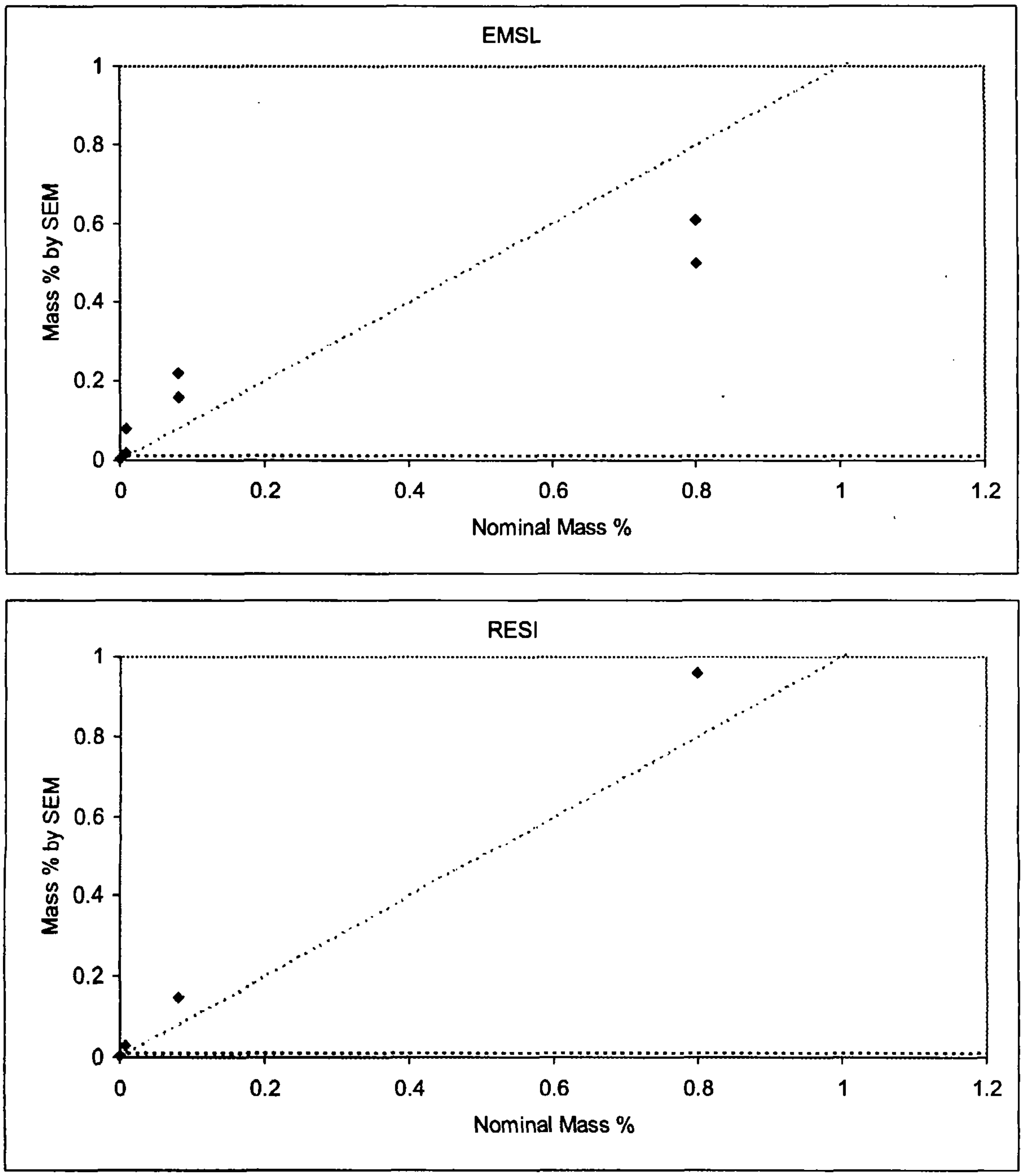

FIGURE 2: INTERLAB COMPARISON OF SEM RESULTS

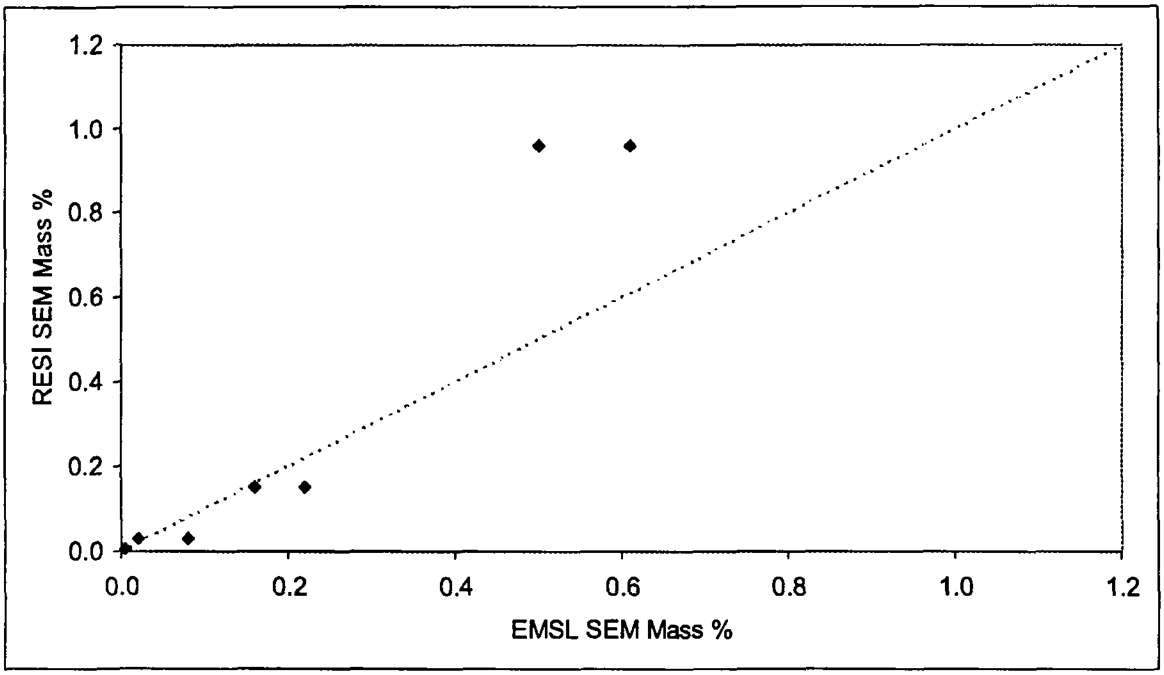

FIGURE 3: IR RESULTS